

09/701751

41- rec'd PCT/PTO 0 1 DEC 2000

PATENT

Attorney Docket No. H 3491 PCT/US**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**CERTIFICATE OF EXPRESS MAIL
(37 C.F.R. § 1.10)

I HEREBY CERTIFY THAT THIS PAPER OR FEE IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE, EXPRESS MAIL POST OFFICE TO ADDRESSEE" UNDER 37 C.F.R. § 1.10, BEARING EXPRESS MAIL LABEL NO. **EK219526757US** ON THIS 1ST DAY OF **DECEMBER, 2000** AND IS ADDRESSED TO: COMMISSIONER FOR PATENTS, WASHINGTON, D.C. 20231


 BY: JAMES M. OLSEN

Int'l Appl. No.: PCT/EP99/03563)

Int'l Filing Date: May 25, 1999)

Priority Date: June 3, 1998)

Applicants: Beatrix KOTTWITZ et al.)

Art Unit: Unassigned

Serial No.: To be assigned)

Examiner: Unassigned

Filed: December 1, 2000)

 For: DETERGENT CONTAINING AMYLASE)
 AND PERCARBONATE)

Commissioner for Patents
Box PCT
Washington, D.C. 20231

TRANSMITTAL OF APPLICATION PAPERS TO U.S.
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. § 371 (37 CFR 1.494 OR 1.495)

This transmittal letter is based upon Form PTO-1390.

The above-identified applicant has filed an International Application under the PCT and hereby submit(s) to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is the FIRST submission of items concerning a filing under 35 U.S.C. §371.

International Application No. PCT/EP99/03563

2. ☐ This is the SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. §371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay.
4. ☒ A proper Demand for International Preliminary Examination (IPE) was made to the appropriate Authority (IPEA) by the 19th month from the earliest claimed priority date (see attached).
5. ☒ A copy of the International Application as amended (35 U.S.C. §371(c)(2)) -
(a) ☐ is transmitted herewith (required when not transmitted by International Bureau).
(b) ☒ has been transmitted by the International Bureau.
(c) ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into the English language is enclosed.
7. ☒ Amendments to the (specification and) claims of the International Application under PCT Article 19 (35 U.S.C. §371 (c)(3))
(a) ☐ are transmitted herewith (required if not transmitted by the International Bureau).
(b) ☐ have been transmitted by the International Bureau.
(c) ☐ have not been made; however, the time limit for making such amendments has NOT expired.
(d) ☒ have not been made and will not be made.
(e) ☐ will be submitted with the appropriate surcharge.
8. ☐ A translation of the amendments to the claims (and/or the specification) under PCT Article 19 (35 U.S.C. §173(c)(3)) is enclosed or will be submitted with the appropriate surcharge.
9. ☒ An unexecuted Oath or Declaration/Power of Attorney of the inventor(s) (35 U.S.C. §371(c)(4)) is enclosed.
10. ☐ A translation of at least the Annexes to the IPE Report under PCT Article 36 (35 U.S.C. §371(c)(5)) is enclosed.

International Application No. PCT/EP99/03563

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98 is enclosed.
12. ☐ An Assignment is enclosed for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31. is included.
13. ☒ A FIRST preliminary amendment is enclosed. **IT IS REQUESTED THAT THE FILING FEES FOR THE CLAIMS BE CALCULATED AFTER THE CLAIM AMENDMENTS IN THE PRELIMINARY AMENDMENT HAVE BEEN ENTERED.**
14. ☐ A substitute specification (including claims, abstract, drawing) is enclosed.
15. ☐ A change of Power of Attorney and/or address letter is enclosed.
16. ☒ Other items of information:
 - ☐ This application is being filed pursuant to 37 CFR 1.494(c) or 1.495(c), and any **missing parts** will be filed before expiration of -
 - ☐ 22 months from the priority date under 37 CFR 1.494(c), or
 - ☐ 32 months from the priority date under 37 CFR 1.495(c).
 - ☐ The undersigned attorney is authorized by the International application and by the inventors to enter the **National Phase** pursuant to 37 CFR 1.494(c) or 1.495(c).

The following additional information relates to the International Application:

- ☒ Receiving Office: EPO (originally filed in the German Patent Office)
- ☐ IPEA (if filing under 37 CFR 1.495): EPO
- ☒ Priority claim(s) (35 USC §§ 119, 365): App. No. 198 24 688.9
Filed: June 3, 1998
- ☒ A copy of the International Search Report is
 - ☒ enclosed.
 - ☒ attached to the copy of the International Application.
- ☒ A copy of the Receiving Office Request Form is enclosed.

International Application No. PCT/EP99/03563

The fee calculation is set forth below.

FEE CALCULATION

[x] A check in payment of the filing fee, calculated as follows, is attached (37 CFR 1.492)

Basic Fee \$860.00

Total Number of claims
in excess of (20) times \$18 \$0.00

Number of independent claims
in excess of (3) times \$80 \$0.00

Fee for multiple dependent
claims \$270 \$0.00

TOTAL FILING FEE \$860.00

Kindly send us the official filing receipt.

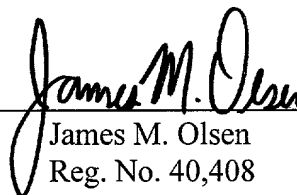
The Commissioner is hereby authorized to charge any additional fees which may be required or to credit any overpayment to Deposit Account No. 03-2775. This is a "general authorization" under 37 CFR 1.25(b), except that no automatic debit of the issue upon allowance is authorized.

Respectfully submitted,

CONNOLLY BOVE LODGE & HUTZ LLP

Date: December 1, 2000

By:


James M. Olsen
Reg. No. 40,408

PATENT
Attorney Docket No. H 3491 PCT/US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Int'l Appl. No.: PCT/EP99/03563)	
)	
Int'l Filing Date: May 25, 1999)	
)	
Priority Date: June 3, 1998)	
)	
Applicants: Beatrix KOTTWITZ et al.)	Art Unit: Unassigned
)	
Serial No.: To be assigned)	Examiner: Unassigned
)	
Filed: December 1, 2000)	
)	
For: DETERGENTS CONTAINING)	
AMYLAASE AND PERCARBONATE)	

**Commissioner for Patents
Box PCT
Washington, D.C. 20231**

Sir:

PRELIMINARY AMENDMENT

Prior to the examination of the above application, please amend this application as follows:

IN THE CLAIMS:

Please cancel claims 2-11, without prejudice or disclaimer of the subject matter thereof.

IN THE ABSTRACT:

Please add the attached Abstract of the Disclosure.

REMARKS

If there are any other fees due in connection with the filing of this response, please charge

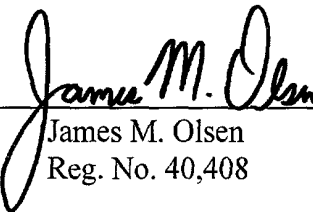
the fees to our Deposit Account No. 03-2775. If a fee is required for an extension of time under 37 C.F.R. § 1.136 not accounted for above, such an extension is requested and the fee should also be charged to our Deposit Account.

Respectfully submitted,

CONNOLLY BOVE LODGE & HUTZ LLP

Dated: December 1, 2000

By:


James M. Olsen
Reg. No. 40,408

2000 DEC 01 10:00 AM

ABSTRACT OF THE DISCLOSURE

An amylase-containing detergent that includes α -amylase from *Bacillus amyloliquefaciens* and alkali metal percarbonate in addition to customary ingredients compatible with such constituents.

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65
66
67
68
69
70
71
72
73
74
75
76
77
78
79
80
81
82
83
84
85
86
87
88
89
90
91
92
93
94
95
96
97
98
99
100
101
102
103
104
105
106
107
108
109
110
111
112
113
114
115
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
131
132
133
134
135
136
137
138
139
140
141
142
143
144
145
146
147
148
149
150
151
152
153
154
155
156
157
158
159
160
161
162
163
164
165
166
167
168
169
170
171
172
173
174
175
176
177
178
179
180
181
182
183
184
185
186
187
188
189
190
191
192
193
194
195
196
197
198
199
200
201
202
203
204
205
206
207
208
209
210
211
212
213
214
215
216
217
218
219
220
221
222
223
224
225
226
227
228
229
230
231
232
233
234
235
236
237
238
239
240
241
242
243
244
245
246
247
248
249
250
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280
281
282
283
284
285
286
287
288
289
290
291
292
293
294
295
296
297
298
299
300
301
302
303
304
305
306
307
308
309
310
311
312
313
314
315
316
317
318
319
320
321
322
323
324
325
326
327
328
329
330
331
332
333
334
335
336
337
338
339
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
361
362
363
364
365
366
367
368
369
370
371
372
373
374
375
376
377
378
379
380
381
382
383
384
385
386
387
388
389
390
391
392
393
394
395
396
397
398
399
400
401
402
403
404
405
406
407
408
409
410
411
412
413
414
415
416
417
418
419
420
421
422
423
424
425
426
427
428
429
430
431
432
433
434
435
436
437
438
439
440
441
442
443
444
445
446
447
448
449
450
451
452
453
454
455
456
457
458
459
460
461
462
463
464
465
466
467
468
469
470
471
472
473
474
475
476
477
478
479
480
481
482
483
484
485
486
487
488
489
490
491
492
493
494
495
496
497
498
499
500
501
502
503
504
505
506
507
508
509
510
511
512
513
514
515
516
517
518
519
520
521
522
523
524
525
526
527
528
529
530
531
532
533
534
535
536
537
538
539
540
541
542
543
544
545
546
547
548
549
550
551
552
553
554
555
556
557
558
559
560
561
562
563
564
565
566
567
568
569
570
571
572
573
574
575
576
577
578
579
580
581
582
583
584
585
586
587
588
589
590
591
592
593
594
595
596
597
598
599
600
601
602
603
604
605
606
607
608
609
610
611
612
613
614
615
616
617
618
619
620
621
622
623
624
625
626
627
628
629
630
631
632
633
634
635
636
637
638
639
640
641
642
643
644
645
646
647
648
649
650
651
652
653
654
655
656
657
658
659
660
661
662
663
664
665
666
667
668
669
670
671
672
673
674
675
676
677
678
679
680
681
682
683
684
685
686
687
688
689
690
691
692
693
694
695
696
697
698
699
700
701
702
703
704
705
706
707
708
709
710
711
712
713
714
715
716
717
718
719
720
721
722
723
724
725
726
727
728
729
730
731
732
733
734
735
736
737
738
739
740
741
742
743
744
745
746
747
748
749
750
751
752
753
754
755
756
757
758
759
760
761
762
763
764
765
766
767
768
769
770
771
772
773
774
775
776
777
778
779
780
781
782
783
784
785
786
787
788
789
790
791
792
793
794
795
796
797
798
799
800
801
802
803
804
805
806
807
808
809
810
811
812
813
814
815
816
817
818
819
820
821
822
823
824
825
826
827
828
829
830
831
832
833
834
835
836
837
838
839
840
841
842
843
844
845
846
847
848
849
850
851
852
853
854
855
856
857
858
859
860
861
862
863
864
865
866
867
868
869
870
871
872
873
874
875
876
877
878
879
880
881
882
883
884
885
886
887
888
889
890
891
892
893
894
895
896
897
898
899
900
901
902
903
904
905
906
907
908
909
910
911
912
913
914
915
916
917
918
919
920
921
922
923
924
925
926
927
928
929
930
931
932
933
934
935
936
937
938
939
940
941
942
943
944
945
946
947
948
949
950
951
952
953
954
955
956
957
958
959
960
961
962
963
964
965
966
967
968
969
970
971
972
973
974
975
976
977
978
979
980
981
982
983
984
985
986
987
988
989
990
991
992
993
994
995
996
997
998
999
1000
1001
1002
1003
1004
1005
1006
1007
1008
1009
1010
1011
1012
1013
1014
1015
1016
1017
1018
1019
1020
1021
1022
1023
1024
1025
1026
1027
1028
1029
1030
1031
1032
1033
1034
1035
1036
1037
1038
1039
1040
1041
1042
1043
1044
1045
1046
1047
1048
1049
1050
1051
1052
1053
1054
1055
1056
1057
1058
1059
1060
1061
1062
1063
1064
1065
1066
1067
1068
1069
1070
1071
1072
1073
1074
1075
1076
1077
1078
1079
1080
1081
1082
1083
1084
1085
1086
1087
1088
1089
1090
1091
1092
1093
1094
1095
1096
1097
1098
1099
1100
1101
1102
1103
1104
1105
1106
1107
1108
1109
1110
1111
1112
1113
1114
1115
1116
1117
1118
1119
1120
1121
1122
1123
1124
1125
1126
1127
1128
1129
1130
1131
1132
1133
1134
1135
1136
1137
1138
1139
1140
1141
1142
1143
1144
1145
1146
1147
1148
1149
1150
1151
1152
1153
1154
1155
1156
1157
1158
1159
1160
1161
1162
1163
1164
1165
1166
1167
1168
1169
1170
1171
1172
1173
1174
1175
1176
1177
1178
1179
1180
1181
1182
1183
1184
1185
1186
1187
1188
1189
1190
1191
1192
1193
1194
1195
1196
1197
1198
1199
1200
1201
1202
1203
1204
1205
1206
1207
1208
1209
1210
1211
1212
1213
1214
1215
1216
1217
1218
1219
1220
1221
1222
1223
1224
1225
1226
1227
1228
1229
1230
1231
1232
1233
1234
1235
1236
1237
1238
1239
1240
1241
1242
1243
1244
1245
1246
1247
1248
1249
1250
1251
1252
1253
1254
1255
1256
1257
1258
1259
1260
1261
1262
1263
1264
1265
1266
1267
1268
1269
1270
1271
1272
1273
1274
1275
1276
1277
1278
1279
1280
1281
1282
1283
1284
1285
1286
1287
1288
1289
1290
1291
1292
1293
1294
1295
1296
1297
1298
1299
1300
1301
1302
1303
1304
1305
1306
1307
1308
1309
1310
1311
1312
1313
1314
1315
1316
1317
1318
1319
1320
1321
1322
1323
1324
1325
1326
1327
1328
1329
1330
1331
1332
1333
1334
1335
1336
1337
1338
1339
1340
1341
1342
1343
1344
1345
1346
1347
1348
1349
1350
1351
1352
1353
1354
1355
1356
1357
1358
1359
1360
1361
1362
1363
1364
1365
1366
1367
1368
1369
1370
1371
1372
1373
1374
1375
1376
1377
1378
1379
1380
1381
1382
1383
1384
1385
1386
1387
1388
1389
1390
1391
1392
1393
1394
1395
1396
1397
1398
1399
1400
1401
1402
1403
1404
1405
1406
1407
1408
1409
1410
1411
1412
1413
1414
1415
1416
1417
1418
1419
1420
1421
1422
1423
1424
1425
1426
1427
1428
1429
1430
1431
1432
1433
1434
1435
1436
1437
1438
1439
1440
1441
1442
1443
1444
1445
1446
1447
1448
1449
1450
1451
1452
1453
1454
1455
1456
1457
1458
1459
1460
1461
1462
1463
1464
1465
1466
1467
1468
1469
1470
1471
1472
1473
1474
1475
1476
1477
1478
1479
1480
1481
1482
1483
1484
1485
1486
1487
1488
1489
1490
1491
1492
1493
1494
1495
1496
1497
1498
1499
1500
1501
1502
1503
1504
1505
1506
1507
1508
1509
1510
1511
1512
1513
1514
1515
1516
1517
1518
1519
1520
1521
1522
1523
1524
1525
1526
1527
1528
1529
1530
1531
1532
1533
1534
1535
1536
1537
1538
1539
1540
1541
1542
1543
1544
1545
1546
1547
1548
1549
1550
1551
1552
1553
1554
1555
1556
1557
1558
1559
1560
1561
1562
1563
1564
1565
1566
1567
1568
1569
1570
1571
1572
1573
1574
1575
1576
1577
1578
1579
1580
1581
1582
1583
1584
1585
1586
1587
1588
1589
1590
1591
1592
1593
1594
1595
1596
1597
1598
1599
1600
1601
1602
1603
1604
1605
1606
1607
1608
1609
1610
1611
1612
1613
1614
1615
1616
1617
1618
1619
1620
1621
1622
1623
1624
1625
1626
1627
1628
1629
1630
1631
1632
1633
1634
1635
1636
1637
1638
1639
1640
1641
1642
1643
1644
1645
1646
1647
1648
1649
1650
1651
1652
1653
1654
1655
1656
1657
1658
1659
1660
1661
1662
1663
1664
1665
1666
1667
1668
1669
1670
1671
1672
1673
1674
1675
1676
1677
1678
1679
1680
1681
1682
1683
1684
1685
1686
1687
1688
1689
1690
1691
1692
1693
1694
1695
1696
1697
1698
1699
1700
1701
1702
1703
1704
1705
1706
1707
1708
1709
1710
1711
1712
1713
1714
1715
1716
1717
1718
1719
1720
1721
1722
1723
1724
1725
1726
1727
1728
1729
1730
1731
1732
1733
1734
1735
1736
1737
1738
1739
1740
1741
1742
1743
1744
1745
1746
1747
1748
1749
1750
1751
1752
1753
1754
1755
1756
1757
1758
1759
1760
1761
1762
1763
1764
1765
1766
1767
1768
1769
1770
1771
1772
1773
1774
1775
1776
1777
1778
1779
1780
1781
1782
1783
1784
1785
1786
1787
1788
1789
1790
1791
1792
1793
1794
1795
1796
1797
1798
1799
1800
1801
1802
1803
1804
1805
1806
1807
1808
1809
1810
1811
1812
1813
1814
1815
1816
1817
1818
1819
1820
1821
1822
1823
1824
1825
1826
1827
1828
1829
1830
1831
1832
1833
1834
1835
1836
1837
1838
1839
1840
1841
1842
1843
1844
1845
1846
1847
1848
1849
1850
1851
1852
1853
1854
1855
1856
1857
1858
1859
1860
1861
1862
1863
1864
1865
1866
1867
1868
1869
1870
1871
1872
1873
1874
1875
1876
1877
1878
1879
1880
1881
1882
1883
1884
1885
1886
1887
1888
1889
1890
1891
1892
1893
1894
1895
1896
1897
1898
1899
1900
1901
1902
1903
1904
1905
1906
1907
1908
1909
1910
1911
1912
1913
1914
1915
1916
1917
1918
1919
1920
1921
1922
1923
1924
1925
1926
1927
1928
1929
1930
1931
1932
1933
1934
1935
1936
1937
1938
1939
1940
1941
1942
1943
1944
1945
1946
1947
1948
1949
1950
1951
1952
1953
1954
1955
1956
1957
1958
1959
1960
1961
1962
1963
1964
1965
1966
1967
1968
1969
1970
1971
1972
1973
1974
1975
1976
1977
1978
1979
1980
1981
1982
1983
1984
1985
1986
1987
1988
1989
1990
1991
1992
1993
1994
1995
1996
1997
1998
1999
2000
2001
2002
2003
2004
2005
2006
2007
2008
2009
2010
2011
2012
2013
2014
2015
2016
2017
2018
2019
2020
2021
2022
2023
2024
2025
2026
2027
2028
2029
2030
2031
2032
2033
2034
2035
2036
2037
2038
2039
2040
2041
2042
2043
2044
2045
2046
2047
2048
2049
2050
2051
2052
2053
2054
2055
2056
2057
2058
2059
2060
2061
2062
2063
2064
2065
2066
2067
2068
2069
2070
2071
2072
2073
2074
2075
2076
2077
2078
2079
2080
2081
2082
2083
2084
2085
2086
2087
2088
2089
2090
2091
2092
2093
2094
2095
2096
2097
2098
2099
2100
2101
2102
2103
2104
2105
2106
2107
2108
2109
2110
2111
2112
2113
2114
2115
2116
2117
2118
2119
2120
2121
2122
2123
2124
2125
2126
2127
2128
2129
2130
2131
2132
2133
2134
2135
2136
2137
2138
2139
2140
2141
2142
2143
2144
2145
2146
2147
2148
2149
2150
2151
2152
2153
2154
2155
2156
2157
2158
2159
2160
2161
2162
2163
2164
2165
2166
2167
2168
2169
2170
2171
2172
2173
2174
2175
2176
2177
2178
2179
2180
2181
2182
2183
2184
2185
2186
2187
2188
2189
2190
2191
2192
2193
2194
2195
2196
2197
2198
2199
2200
2201
2202
2203
2204
2205
2206
2207
2208
2209
2210
2211
2212
2213
2214
2215
2216
2217
2218
2219
2220
2221
2222
2

WO 99/63036

Detergents comprising amylase and percarbonate

The present invention relates to enzyme containing
detergents comprising besides customary constituents an
5 amylase from *Bacillus amyloliquefaciens* and a certain
peroxidic bleach.

Laundry detergents, in addition to the ingredients
vital to the laundering process, such as surfactants
10 and builder materials, generally comprise further
constituents, which may be grouped together under the
heading of wash auxiliaries and which embrace such
different active substance groups as foam regulators,
graying inhibitors, bleaches, bleach activators, and
15 color transfer inhibitors. Such auxiliaries also
include substances which assist the surfactant
performance by means of enzymatic degradation of stains
present on the textile. The same applies, mutatis
mutandis, to cleaning products for hard surfaces. As
20 well as the proteases, which assist in removing
proteins, and the fat-cleaving lipases, particular
importance in this context attaches to the amylases.
Amylases have the function of facilitating the removal
of starchy stains by means of catalytic hydrolysis of
25 the starch polysaccharide, and have been used for this
purpose for a fairly long time in dishwashing
detergents, but also in detergents for use in textile
laundering. In by far the great majority of cases the
amylase involved has comprised a heat-stable amylase
30 from *Bacillus licheniformis*, which is customary in
commerce under the designation Termamyl®, for example.
More recently, there has been increased use in such
compositions of genetically manipulated amylases; that
is, amylases whose amino acid sequence has been
35 altered, using gene technology methods, in comparison
to naturally occurring amylases. As well as increasing
their capacity to perform, the objective of genetically
modifying amylases is essentially to enhance the
stability of the enzyme, especially their stability to

attack by oxidizing agents. One approach toward achieving this objective, which was proposed in International Patent Application WO 94/18314, consists in removing particularly oxidation-susceptible amino acids, such as methionine, tryptophan, cysteine or tyrosine, from the amino acid sequence of the amylase, or replacing them by other amino acids more stable to oxidation. A similar approach is proposed in International Patent Application WO 95/21247, which recommends replacing at least one methionine in the amylase amino acid sequence by an amino acid which is neither methionine nor cysteine.

Although such genetic modifications may lead to improved amylase stability under certain application conditions, they do not help to increase the contribution of the amylase to the wash or cleaning performance of corresponding compositions comprising the amylase.

It has surprisingly now been found that the combination of a naturally occurring α -amylase with a certain peroxidic oxidizing agent leads to unexpectedly synergistic performance improvements when used in detergents.

The invention accordingly provides an amylase containing detergent which comprises α -amylase from *Bacillus amyloliquefaciens* and an alkali metal percarbonate, as well as customary ingredients compatible with such constituents.

The invention further provides for the use of such a combination for enhancing the cleaning performance of detergents, especially with respect to starchy and/or colored stains when used in detergent solutions, especially aqueous detergent solutions. In this context, cleaning performance with respect to colored stains is to be understood in its widest context and

embraces not only the bleaching of soil present on the textile and the bleaching of soil which is in the wash liquor, having become detached from the textile, but also the oxidative destruction of textile colors present in the wash liquor, having become detached from textiles under the wash conditions, before they are able to attach to different-colored textiles. Also, in the context of use in cleaning solutions for hard surfaces, this term refers both to the bleaching of soil present on the hard surface, especially tea, and to the bleaching of soil which is present in the dishwashing liquor, having become detached from the hard surface.

α -Amylase from *Bacillus amyloliquefaciens* has been known for a long time, for example, from the U.S. Patent US 1 227 374. It is available commercially, for example, under the designation Amylase BAN®.

A composition of the invention contains preferably from 0.001 mg to 0.5 mg, in particular from 0.02 mg to 0.3 mg, of amylolytic protein per gram of the overall composition. The protein concentration may be determined using known methods, such as the bicinchonic acid technique (BCA technique, Pierce Chemical Co., Rockford, IL) or the Biuret technique (A.G. Gornall, C.S. Bardawill and M.M. David, J. Biol. Chem. 177, 751-766, 1948).

A composition of the invention comprises preferably up to 50% by weight, in particular from 5% by weight to 30% by weight, of alkali metal percarbonate, sodium percarbonate being particularly preferred. It can be prepared by known processes and, especially for use in particulate compositions, may if desired be compounded in granular form and/or stabilized and/or coated, as known, for example, from International Patent Applications WO 91/15423, WO 92/17400, WO 92/17404, WO 93/04159, WO 93/04982, WO 93/20007, WO 94/03553,

WO 94/05594, WO 94/14701, WO 94/14702, WO 94/24044,
WO 95/02555, WO 95/02672, WO 95/06615, WO 95/15291 or
WO 95/15292 or in European Patent Applications
EP 0 459 625, EP 0 487 256, EP 0 567 140, EP 0 623 553,
5 EP 0 592 969 or EP 0 748 764. For reasons of stability
it is preferably used in the form of granules which
have been prepared from and/or coated with the aid of
alkaline earth metal sulfate, alkali metal sulfate,
alkali metal silicate, alkaline earth metal halide,
10 alkali metal halide, alkali metal carbonate, alkali
metal hydrogencarbonate, alkali metal phosphate, alkali
metal borate, alkali metal perborate, boric acid,
partially hydrated aluminosilicate, carboxylic acids,
dicarboxylic acids, polymers of unsaturated carboxylic
15 and/or dicarboxylic acids, or mixtures of these. In one
preferred embodiment it has a morphology index (MI), as
defined in EP 0 451 893, of below 0.06.

In one preferred embodiment of compositions of the
20 invention, the ratio of α -amylase to alkali metal
percarbonate is in the range from 0.0001 mg to 0.1 mg,
in particular from 0.001 mg to 0.01 mg, of amylolytic
protein per % by weight of alkali metal percarbonate in
the detergent.

25 In addition to the active substance combination used in
accordance with the invention, the detergents of the
invention, which may be present as solids, especially
pulverulent solids, in postcompacted particle form, as
30 homogeneous solutions or suspensions may in principle
comprise all known ingredients which are customary in
such compositions. The compositions of the invention
may in particular comprise builder substances,
surfactants, additional bleaches based on organic
35 and/or inorganic peroxygen compounds, bleach
activators, water miscible organic solvents, additional
enzymes, sequesterants, electrolytes, pH regulators,
and further auxiliaries, such as optical brighteners,
graying inhibitors, color transfer inhibitors, foam

regulators, silver corrosion inhibitors, and also dyes and fragrances.

5 The compositions of the invention may comprise a surfactant or two or more surfactants, suitable surfactants comprising in particular anionic surfactants, nonionic surfactants and mixtures thereof, but also cationic, zwitterionic and amphoteric surfactants.

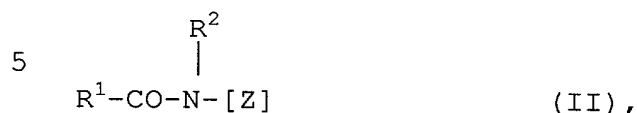
10

Suitable nonionic surfactants are, in particular, alkyl glycosides and ethoxylation and/or propoxylation products of alkyl glycosides or linear or branched alcohols having in each case 12 to 18 carbon atoms in the alkyl moiety and from 3 to 12, preferably from 4 to 10, alkyl ether groups. It is also possible to use corresponding ethoxylation and/or propoxylation products of N-alkylamines, vicinal diols, fatty acid esters and fatty acid amides, which in terms of the 15 alkyl moiety correspond to the aforementioned long chain alcohol derivatives, and also alkylphenols having 5 to 12 carbon atoms in the alkyl radical.

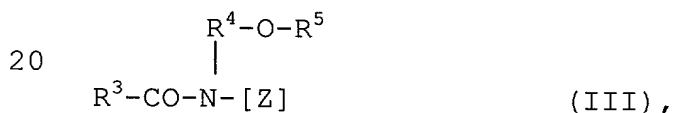
20 Nonionic surfactants used are preferably alkoxyated, advantageously ethoxylated, especially primary, alcohols having preferably 8 to 18 carbon atoms and on average from 1 to 12 mol of ethylene oxide (EO) per mole of alcohol, in which the alcohol radical may be linear or, preferably, methyl-branched in position 2 25 and/or may comprise linear and methyl-branched radicals in a mixture, as are commonly present in oxo alcohol radicals. In particular, however, preference is given to alcohol ethoxylates containing linear radicals from alcohols of natural origin having 12 to 18 carbon 30 atoms, e.g., from coconut, palm, tallow fatty or oleyl alcohol and on average from 2 to 8 EO per mole of alcohol. Preferred ethoxylated alcohols include, for example, C₁₂₋₁₄ alcohols containing 3 EO or 4 EO, C₉₋₁₁ alcohols containing 7 EO, C₁₃₋₁₅ alcohols containing 35

3 EO, 5 EO, 7 EO or 8 EO, C₁₂₋₁₈ alcohols containing 3 EO, 5 EO or 7 EO, and mixtures thereof, such as mixtures of C₁₂₋₁₄ alcohol containing 3 EO and C₁₂₋₁₈ alcohol containing 7 EO. The stated degrees of
5 ethoxylation represent statistical mean values, which for a specific product may be an integer or a fraction. Preferred alcohol ethoxylates have a narrowed homolog distribution (narrow range ethoxylates, NREs). In addition to these nonionic surfactants it is also
10 possible to use fatty alcohols containing more than 12 EO. Examples thereof are (tallow) fatty alcohols containing 14 EO, 16 EO, 20 EO, 25 EO, 30 EO or 40 EO. Especially in detergents for use in machine dishwashing processes, it is common to use extremely low-foaming
15 compounds. These include, preferably, C_{12-C18} alkyl polyethylene glycol-polypropylene glycol ethers having in each case up to 8 mol of ethylene oxide and propylene oxide units in the molecule. However, it is also possible to use other nonionic surfactants which
20 are known to be low-foaming, such as, for example, C_{12-C18} alkyl polyethylene glycol-polybutylene glycol ethers having in each case up to 8 mol of ethylene oxide and butylene oxide units in the molecule, and also endgroup-capped alkyl polyalkylene glycol mixed ethers.
25 Particular preference is also given to the hydroxyl-containing alkoxyated alcohols as are described in European Patent Application EP 0 300 305, referred to as hydroxy mixed ethers. The nonionic surfactants also include alkyl glycosides of the general formula RO(G)_x,
30 where R is a primary straight-chain or methyl-branched aliphatic radical, especially an aliphatic radical methyl-branched in position 2, containing 8 to 22, preferably 12 to 18, carbon atoms, and G represents a glucose unit having 5 or 6 carbon atoms, preferably
35 glucose. The degree of oligomerization, x, which indicates the distribution of monoglycosides and oligoglycosides, is any desired number - which, as a variable to be determined analytically, may also be a fraction - between 1 and 10; preferably, x is from 1.2

to 1.4. Further suitable surfactants are polyhydroxy fatty acid amides of the formula (II)



where R^1CO is an aliphatic acyl radical having 6 to 22 carbon atoms, R^2 is hydrogen or an alkyl or hydroxyalkyl radical having 1 to 4 carbon atoms, and [Z] is a linear or branched polyhydroxyalkyl radical having 3 to 10 carbon atoms and from 3 to 10 hydroxyl groups. The polyhydroxy fatty acid amides are derived preferably from reducing sugars having 5 or 6 carbon atoms, especially glucose. The group of the polyhydroxy fatty acid amides also includes compounds of the formula (III)



where R^3 is a linear or branched alkyl or alkenyl radical having 7 to 12 carbon atoms, R^4 is a linear, branched or cyclic alkylene radical or an arylene radical having 2 to 8 carbon atoms and R^5 is a linear, branched or cyclic alkyl radical or an aryl radical or an oxyalkyl radical having 1 to 8 carbon atoms, preference being given to $\text{C}_1\text{-C}_4$ alkyl radicals or phenyl radicals, and [Z] is a linear polyhydroxyalkyl radical whose alkyl chain is substituted by at least two hydroxyl groups, or alkoxyated, preferably ethoxyated or propoxyated, derivatives of said radical. Here too, [Z] is preferably obtained by reductive amination of a sugar such as glucose, fructose, maltose, lactose, galactose, mannose, or xylose. The N-alkoxy- or N-aryloxy-substituted compounds may then be converted to the desired polyhydroxy fatty acid amides, for example, in accordance with the teaching of International Patent Application WO 95/07331 by

reaction with fatty acid methyl esters in the presence of an alkoxide as catalyst. A further class of nonionic surfactants used with preference, which are used either as sole nonionic surfactant or in combination with other nonionic surfactants, in particular together with alkoxyated fatty alcohols and/or alkyl glycosides, are alkoxyated, preferably ethoxyated, or ethoxyated and propoxyated, fatty acid alkyl esters, preferably having 1 to 4 carbon atoms in the alkyl chain, especially fatty acid methyl esters, as are described, for example, in Japanese Patent Application JP 58/217598, or those prepared preferably by the process described in International Patent Application WO 90/13533. Nonionic surfactants of the amine oxide type, examples being N-cocoalkyl-N,N-dimethylamine oxide and N-tallowalkyl-N,N-dihydroxyethylamine oxide, and of the fatty acid alkanolamide type, may also be suitable. The amount of these nonionic surfactants is preferably not more than that of the ethoxyated fatty alcohols, in particular not more than half thereof. Further suitable surfactants include those known as gemini surfactants. This term is used generally to refer to those compounds which possess two hydrophilic groups per molecule. These groups are generally separated from one another as a result of what is known as a spacer. This spacer is generally a carbon chain, which should be long enough to give the hydrophilic groups a sufficient spacing to allow them to act independently of one another. Surfactants of this kind are generally notable for an unusually low critical micelle concentration and the ability to reduce greatly the surface tension of water. In exceptional cases, however, the expression gemini surfactants is used to embrace not only dimeric but also, correspondingly, trimeric surfactants. Examples of suitable gemini surfactants are sulfated hydroxy mixed ethers in accordance with German Patent Application DE 43 21 022 or dimer alcohol bis- and trimer alcohol tris-sulfates and ether sulfates in accordance with German Patent

Application DE 195 03 061. Endgroup-capped dimeric and trimeric mixed ethers in accordance with German Patent Application DE-A-195 13 391 are notable in particular for their bi- and multifunctionality. Thus said
5 endgroup-capped surfactants possess good wetting properties and are low-foaming, so making them particularly suitable for use in machine washing or cleaning processes. However, it is also possible to use
10 gemini-polyhydroxy fatty acid amides or polypolyhydroxy fatty acid amides, as described in International Patent Applications WO 95/19953, WO 95/19954, and WO 95/19955.

Suitable anionic surfactants are, in particular, soaps and those containing sulfate or sulfonate groups.
15 Preferred surfactants of the sulfonate type are C₉₋₁₃ alkylbenzenesulfonates, olefinsulfonates, i.e., mixtures of alkenesulfonates and hydroxyalkanesulfonates, and also disulfonates, as are obtained, for example, from C₁₂₋₁₈ monoolefins having a terminal or
20 internal double bond by sulfonating with gaseous sulfur trioxide followed by alkaline or acidic hydrolysis of the sulfonation products. Also suitable are alkanesulfonates, which are obtained from C₁₂₋₁₈ alkanes, for example, by sulfochlorination or sulfoxidation with
25 subsequent hydrolysis or neutralization, respectively. Likewise suitable, in addition, are the esters of α -sulfo fatty acids (ester sulfonates), e.g., the α -sulfonated methyl esters of hydrogenated coconut, palm kernel or tallow fatty acids, which are prepared
30 by α -sulfonation of the methyl esters of fatty acids of plant and/or animal origin having 8 to 20 carbon atoms in the fatty acid molecule, followed by neutralization to give water-soluble mono-salts. Preferably, these comprise the α -sulfonated esters of hydrogenated
35 coconut, palm, palm kernel or tallow fatty acids, it being possible as well for sulfonation products of unsaturated fatty acids, e.g. oleic acid, to be present in small amounts, preferably in amounts of not more than about 2 to 3% by weight. Particular preference is

given to α -sulfo fatty acid alkyl esters having an alkyl chain of not more than 4 carbon atoms in the ester group, examples being methyl esters, ethyl esters, propyl esters, and butyl esters. With particular advantage, the methyl esters of the α -sulfo fatty acids (MES) are used, and also their saponified di-salts. Further suitable anionic surfactants are sulfated fatty acid glycerol esters which are the monoesters, diesters and triesters, and mixtures thereof, as obtained in the preparation by esterification of a monoglycerol with from 1 to 3 mol of fatty acid or in the transesterification of triglycerides with from 0.3 to 2 mol of glycerol. Preferred alk(en)yl sulfates are the alkali metal salts, and especially the sodium salts, of the sulfuric monoesters of C₁₂-C₁₈ fatty alcohols, examples being those of coconut fatty alcohol, tallow fatty alcohol, lauryl, myristyl, cetyl or stearyl alcohol, or of C₁₀-C₂₀ oxo alcohols, and those monoesters of secondary alcohols of this chain length. Preference is also given to alk(en)yl sulfates of said chain length which contain a synthetic straight-chain alkyl radical prepared on a petrochemical basis, these sulfates possessing degradation properties similar to those of the corresponding compounds based on fatty-chemical raw materials. From a detergents standpoint, C₁₂-C₁₆ alkyl sulfates and C₁₂-C₁₅ alkyl sulfates, and also C₁₄-C₁₅ alkyl sulfates, are particularly preferred. In addition, 2,3-alkyl sulfates, which may for example be prepared in accordance with US Patents 3,234,258 or 5,075,041 and obtained as commercial products from Shell Oil Company under the name DAN®, are suitable anionic surfactants. Also suitable are the sulfuric monoesters of the straight-chain or branched C₇₋₂₁ alcohols ethoxylated with from 1 to 6 mol of ethylene oxide, such as 2-methyl-branched C₉₋₁₁ alcohols containing on average 3.5 mol of ethylene oxide (EO) or C₁₂₋₁₈ fatty alcohols containing from 1 to 4 EO. Preferred anionic surfactants further include the salts

of alkylsulfosuccinic acid, which are also referred to as sulfosuccinates or as sulfosuccinic esters and which constitute the monoesters and/or diesters of sulfosuccinic acid with alcohols, preferably fatty alcohols and especially ethoxylated fatty alcohols. Preferred sulfosuccinates comprise C₈₋₁₈ fatty alcohol radicals or mixtures thereof. Especially preferred sulfosuccinates contain a fatty alcohol radical derived from ethoxylated fatty alcohols which themselves represent nonionic surfactants. Particular preference is given in turn to sulfosuccinates whose fatty alcohol radicals are derived from ethoxylated fatty alcohols having a narrowed homolog distribution. Similarly, it is also possible to use alk(en)ylsuccinic acid containing preferably 8 to 18 carbon atoms in the alk(en)yl chain, or salts thereof. Further suitable anionic surfactants include fatty acid derivatives of amino acids, for example, of N-methyltaurine (taurides) and/or of N-methylglycine (sarcosides). Particular preference is given here to sarcosides and to the sarcosinates and, of these, especially the sarcosinates of higher fatty acids, which may be mono- or polyunsaturated, such as oleyl sarcosinate. Further suitable anionic surfactants are, in particular, soaps. Suitable soaps include in particular saturated fatty acid soaps, such as the salts of lauric acid, myristic acid, palmitic acid, stearic acid, hydrogenated erucic acid and behenic acid, and, in particular, mixtures of soaps derived from natural fatty acids, e.g., coconut, palm kernel, or tallow fatty acids. Together with these soaps, or as substitutes for soaps, it is also possible to use the known alkenylsuccinic salts.

The anionic surfactants, including the soaps, may be present in the form of their sodium, potassium or ammonium salts and also as soluble salts of organic bases, such as mono-, di- or triethanolamine. Preferably, the anionic surfactants are in the form of

their sodium or potassium salts, in particular in the form of the sodium salts.

In laundry detergents of the invention, surfactants are present in proportions of preferably from 5% by weight to 50% by weight, in particular from 8% by weight to 30% by weight, whereas compositions for cleaning hard surfaces, especially for the machine cleaning of kitchen- and tableware, have lower surfactant contents of up to 10% by weight, in particular up to 5% by weight, and preferably in the range from 0.5% by weight to 3% by weight.

A composition of the invention comprises preferably at least one water soluble and/or water insoluble, organic and/or inorganic builder. The water soluble organic builder substances include polycarboxylic acids, especially citric acid and sugar acids, monomeric and polymeric amino polycarboxylic acids, in particular methylglycinediacetic acid, nitrilotriacetic acid and ethylenediaminetetraacetic acid, and also polyaspartic acid, polyphosphonic acids, especially aminotris-(methylenephosphonic acid), ethylenediaminetetrakis-(methylenephosphonic acid) and 1-hydroxyethane-1,1-diphosphonic acid, polymeric hydroxy compounds such as dextrin, and also polymeric (poly)carboxylic acids, especially the polycarboxylates of European Patent EP 0 625 992 or of International Patent Application WO 92/18542 or of European Patent EP 0 232 202, which are obtainable by oxidizing polysaccharides or, respectively, dextrans; polymeric acrylic acids, methacrylic acids, maleic acids and copolymers thereof, which may also contain in copolymerized form small fractions of polymerizable substances without carboxylic acid functionality. The relative molecular mass of the homopolymers of unsaturated carboxylic acids is generally between 3000 and 200,000, that of the copolymers between 2000 and 200,000, preferably from 30,000 to 120,000, based in each case on free

acid. A particularly preferred acrylic acid-maleic acid copolymer has a relative molecular mass of from 30,000 to 100,000. Commercially customary products are, for example, Sokalan® CP 5, CP 10 and PA 30 from BASF.

5 Suitable though less preferred compounds of this class are copolymers of acrylic acid or methacrylic acid with vinyl ethers, such as vinyl methyl ethers, vinyl esters, ethylene, propylene and styrene, in which the acid fraction is at least 50% by weight. As water
10 soluble organic builder substances it is also possible to use terpolymers containing as monomers two unsaturated acids and/or their salts and, as the third monomer, vinyl alcohol and/or an esterified vinyl alcohol or a carbohydrate. The first acidic monomer or
15 salt thereof is derived from a monoethylenically unsaturated C₃-C₈ carboxylic acid and, preferably, from a C₃-C₄ monocarboxylic acid, especially from (meth)acrylic acid. The second acidic monomer or salt thereof may be a derivative of a C₄-C₈ dicarboxylic
20 acid, maleic acid being particularly preferred, and/or a derivative of an allylsulfonic acid substituted in position 2 by an alkyl or aryl radical. Such polymers may be prepared in particular by processes which are described in German Patent DE 42 21 381 and German
25 Patent Application DE 43 00 772, and generally have a relative molecular mass of between 1000 and 200,000. Further preferred copolymers are those described in German Patent Applications DE 43 03 320 and DE 44 17 734, containing as monomers preferably
30 acrolein and acrylic acid/acrylic acid salts, and/or vinyl acetate. The organic builder substances, especially for the preparation of liquid compositions, may be used in the form of aqueous solutions, preferably aqueous solutions with a strength of from 30
35 to 50 percent by weight. All said acids are generally used in the form of their water soluble salts, especially their alkali metal salts.

Organic builder substances of this kind may be present, if desired, in amounts of up to 40% by weight, in particular up to 25% by weight, and preferably from 1% by weight to 8% by weight. Amounts close to the stated upper limit are used preferably in pastelike or liquid compositions of the invention, especially those containing water.

Suitable water soluble inorganic builder materials include, in particular, alkali metal silicates, alkali metal carbonates and alkali metal phosphates, which may be present in the form of their alkaline, neutral or acidic sodium or potassium salts. Examples thereof are trisodium phosphate, tetrasodium diphosphate, disodium dihydrogen diphosphate, pentasodium triphosphate, so-called sodium hexametaphosphate, oligomeric trisodium phosphate having degrees of oligomerization from 5 to 1000, especially from 5 to 50, and also the corresponding potassium salts and/or mixtures of sodium and potassium salts. Water insoluble, water dispersible inorganic builder materials used are, in particular, crystalline or amorphous alkali metal aluminosilicates, in amounts of up to 50% by weight, preferably not more than 40% by weight, and in liquid compositions in particular from 1% by weight to 5% by weight. Among these, preference is given to the crystalline sodium aluminosilicates of detergent quality, especially zeolite A, P and, if appropriate, X, alone or in mixtures, in the form for example of a cocrystallizate of zeolites A and X (Vegobond® AX, a commercial product from Condea Augusta S.p.A.). Amounts close to the stated upper limit are used preferably in solid, particulate compositions. Suitable aluminosilicates possess in particular no particles having a size of more than 30 μm , and preferably consist at least 80% by weight of particles having a size below 10 μm . Their calcium binding capacity, which may be determined in accordance with the information in German Patent DE 24 12 837, is

generally in the range from 100 to 200 mg of CaO per gram.

Suitable substitutes or partial substitutes for said aluminosilicate are crystalline alkali metal silicates, which may be present alone or in a mixture with amorphous silicates. The alkali metal silicates which can be used as builders in the compositions of the invention preferably have a molar ratio of alkali metal oxide to SiO_2 of below 0.95, in particular from 1:1.1 to 1:12, and may be present in amorphous or crystalline form. Preferred alkali metal silicates are the sodium silicates, especially the amorphous sodium silicates, having a molar ratio $\text{Na}_2\text{O}:\text{SiO}_2$ of from 1:2 to 1:2.8. Those with a molar ratio $\text{Na}_2\text{O}:\text{SiO}_2$ of from 1:1.9 to 1:2.8 may be prepared by the process of European Patent Application EP 0 425 427. As crystalline silicates which may be present alone or in a mixture with amorphous silicates it is preferred to use crystalline phyllosilicates of the general formula $\text{Na}_2\text{Si}_x\text{O}_{2x+1} \cdot y\text{H}_2\text{O}$, where x, the so-called modulus, is a number from 1.9 to 22, in particular from 1.9 to 4, and y is a number from 0 to 33, and preferred values for x are 2, 3 or 4. Crystalline phyllosilicates which fall under this general formula are described, for example, in European Patent Application EP 0 164 514. Preferred crystalline phyllosilicates are those where x in the stated general formula adopts the values 2 or 3. In particular, both β - and δ -sodium disilicates ($\text{Na}_2\text{Si}_2\text{O}_5 \cdot y\text{H}_2\text{O}$) are preferred, with β -sodium disilicate, for example, being obtainable by the process described in International Patent Application WO 91/08171. δ -Sodium silicates having a modulus of between 1.9 and 3.2 may be prepared in accordance with Japanese Patent Applications JP 04/238 809 and JP 04/260 610. In addition, virtually anhydrous crystalline alkali metal silicates of the abovementioned general formula wherein x is a number from 1.9 to 2.1, prepared from amorphous alkali metal silicates, and preparable as described in European

Patent Applications EP 0 548 599, EP 0 502 325 and EP 0 452 428, may be used in compositions of the invention. In a further preferred embodiment of compositions of the invention, a crystalline sodium phyllosilicate having a modulus of from 2 to 3 is used, as may be prepared from sand and soda by the process of European Patent Application EP 0 436 835. Crystalline sodium silicates having a modulus in the range from 1.9 to 3.5, as are obtainable by the processes of European Patents EP 0 164 552 and/or EP 0 294 753, are used in a further preferred embodiment of compositions of the invention. Crystalline sheetlike silicates of the abovementioned formula are sold by Clariant GmbH under the trade name Na-SKS, e.g., Na-SKS-1 ($\text{Na}_2\text{Si}_{22}\text{O}_{45} \cdot x\text{H}_2\text{O}$, kenyaite), Na-SKS-2 ($\text{Na}_2\text{Si}_{14}\text{O}_{29} \cdot x\text{H}_2\text{O}$, magadiite), Na-SKS-3 ($\text{Na}_2\text{Si}_8\text{O}_{17} \cdot x\text{H}_2\text{O}$) or Na-SKS-4 ($\text{Na}_2\text{Si}_4\text{O}_9 \cdot x\text{H}_2\text{O}$, makatite). Of these, those particularly suitable include Na-SKS-5 ($\alpha\text{-Na}_2\text{Si}_2\text{O}_5$), Na-SKS-7 ($\beta\text{-Na}_2\text{Si}_2\text{O}_5$, natrosilite), Na-SKS-9 ($\text{NaHSi}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$), Na-SKS-10 ($\text{NaHSi}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$, kanemite), Na-SKS-11 ($t\text{-Na}_2\text{Si}_2\text{O}_5$) and Na-SKS-13 (NaHSi_2O_5), but especially Na-SKS-6 ($\delta\text{-Na}_2\text{Si}_2\text{O}_5$). A review of crystalline phyllosilicates is given, for example, by the articles published in "Hoechst High Chem Magazin 14/1993" on pages 33-38 and in "Seifen-Öle-Fette-Wachse, Vol. 116, No. 20/1990" on pages 805-808. In one preferred embodiment of compositions of the invention, use is made of a granular compound of crystalline phyllosilicate and citrate, of crystalline phyllosilicate and abovementioned (co)polymeric polycarboxylic acid, as is described, for example, in German Patent Application DE 198 19 187, or of alkali metal silicate and alkali metal carbonate, as is described, for example, in International Patent Application WO 95/22592 or as is available commercially, for example, under the name Nabion® 15.

Builder substances may be present in the compositions of the invention, if desired, in amounts of up to 90% by weight. They are preferably present in amounts of up

to 75% by weight. Laundry detergents of the invention have builder contents of in particular from 5% by weight to 50% by weight. In compositions of the invention for cleaning hard surfaces, especially for the machine cleaning of kitchen- and tableware, the amount of builder substances is in particular from 5% by weight to 88% by weight, preferably no water insoluble builder materials being used in such compositions. In a preferred embodiment of compositions of the invention intended in particular for the machine washing of kitchen- and tableware, from 20% by weight to 40% by weight of water soluble organic builder, especially alkali metal citrate, from 5% by weight to 15% by weight of alkali metal carbonate, and from 20% by weight to 40% by weight of alkali metal disilicate are present.

Additional peroxygen compounds that may be suitable for use in compositions of the invention include, in particular, organic peracids or peracidic salts of organic acids, such as phthalimidopercaproic acid, perbenzoic acid or salts of diperdodecanedioic acid, hydrogen peroxide, and inorganic salts which give off hydrogen peroxide under the washing conditions, including perborate, percarbonate, persilicate and/or persulfate such as Caroate. Where solid peroxygen compounds are additionally to be used, they may be employed in the form of powders or granules, which may also have been coated in a manner which is known in principle. The addition of small amounts of known bleach stabilizers such as, for example, of phosphonates, borates and/or metaborates and metasilicates, and also magnesium salts such as magnesium sulfate, may also be judicious here.

Bleach activators which may be used are compounds which under perhydrolysis conditions give rise to aliphatic peroxo carboxylic acids having preferably 1 to 10 carbon atoms, in particular 2 to 4 carbon atoms, and/or

substituted or unsubstituted perbenzoic acid. Suitable substances are those which carry O-acyl and/or N-acyl groups of the stated number of carbon atoms, and/or substituted or unsubstituted benzoyl groups. Preference is given to polyacylated alkylenediamines, especially tetraacetyleneethylenediamine (TAED), acylated triazine derivatives, especially 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine (DADHT), acylated glycolurils, especially tetraacetylglycoluril (TAGU), N-acyl imides, especially N-nonanoylsuccinimide (NOSI), acylated phenolsulfonates, especially n-nonanoyl- or isononanoyloxybenzenesulfonate (n- or iso-NOBS), carboxylic anhydrides, especially phthalic anhydride, acylated polyhydric alcohols, especially triacetin, ethylene glycol diacetate, 2,5-diacetoxy-2,5-dihydrofuran, and the enol esters known from German Patent Applications DE 196 16 693 and DE 196 16 767, and also acetylated sorbitol and mannitol and/or the mixtures thereof (SORMAN) described in European Patent Application EP 0 525 239, acylated sugar derivatives, especially pentaacetylglucose (PAG), pentaacetylfructose, tetraacetylxylose and octaacetyllactose, and acetylated, optionally N-alkylated glucamine and gluconolactone, and/or N-acylated lactams, for example, N-benzoylcaprolactam, which are known from International Patent Applications WO 94/27970, WO 94/28102, WO 94/28103, WO 95/00626, WO 95/14759 and WO 95/17498. The hydrophilically substituted acylacetals known from German Patent Application DE 196 16 769 and acyllactams described in German Patent Application DE 196 16 770 and in International Patent Application WO 95/14075 are likewise used with preference. The combinations of conventional bleach activators known from German Patent Application DE 44 43 177, may also be used. Such bleach activators may, especially when the abovementioned hydrogen peroxide donating bleaches are present, be present in customary quantities, preferably in amounts of from 0.5% by weight to 10% by weight, and in particular from

1% by weight to 8% by weight, based on overall composition, but are preferably all absent when the percarboxylic acid essential to the invention is used as the sole bleach.

5

In addition to the conventional bleach activators or instead of them it is also possible for the sulfonimines and/or bleach boosting transition metal salts or transition metal complexes, known from
10 European Patents EP 0 446 982 and EP 0 453 003, to be present as so-called bleaching catalysts, preferably in an amount of up to 1% by weight, in particular from 0.001% by weight to 0.25% by weight.

15

Enzymes which may be used in the compositions in addition to the amylase that is essential to the invention include those from the class of the proteases, lipases, cutinases, pullulanases, hemicellulases, cellulases, oxidases, laccases and
20 peroxidases, and mixtures thereof. If desired, amylases other than the amylase essential to the invention may be present in addition to it. Particularly suitable active enzymatic substances are those obtained from fungi or bacteria, such as *Bacillus subtilis*, *Bacillus licheniformis*, *Bacillus lentus*, *Streptomyces griseus*, *Humicola lanuginosa*, *Humicola insolens*, *Pseudomonas pseudoalcaligenes*, *Pseudomonas cepacia* or *Coprinus cinereus*. The amylase essential to the invention and the enzymes that may be used in addition may - as
25 described, for example, in European Patent EP 0 564 476 or in International Patent Application WO 94/23005 - be adsorbed on carrier substances and/or embedded in coating substances in order to protect them against premature inactivation. In the detergents of the
30 invention they are present preferably in amounts of up to 5% by weight, in particular from 0.2% by weight to 4% by weight. Where the composition of the invention comprises protease, it preferably has a proteolytic activity in the range from about 100 PU/g to about
35

10,000 PU/g, in particular from 300 PU/g to 8000 PU/g. Where two or more enzymes are to be used in the composition of the invention, this may be accomplished by incorporating the two or more separate, or
5 conventionally separately compounded enzymes, or by means of two or more enzymes compounded together in granules, as known, for example, from International Patent Applications WO 96/00772 or WO 96/00773.

10 The organic solvents which may be used besides water in the compositions of the invention, especially if they are present in liquid or paste form, include alcohols having 1 to 4 carbon atoms, especially methanol, ethanol, isopropanol and tert-butanol, diols having 2
15 to 4 carbon atoms, especially ethylene glycol and propylene glycol, and also mixtures thereof and the ethers derivable from the aforementioned classes of compound. Water miscible solvents of this kind are present in the compositions of the invention preferably
20 in amounts of not more than 30% by weight, in particular from 6% by weight to 20% by weight.

Additionally, the compositions may comprise further constituents customary in detergents. These optional
25 constituents include, in particular, enzyme stabilizers, graying inhibitors, color transfer inhibitors, foam inhibitors, and optical brighteners and also dyes and fragrances. In order to protect against silver corrosion, silver corrosion inhibitors
30 may be used in dishwashing detergents of the invention. A cleaning product of the invention for hard surfaces may, furthermore, comprise abrasive constituents, especially from the group consisting of quartz flours, wood flours, polymer flours, chalks and glass
35 microbeads, and mixtures thereof. Abrasives are present in the detergents of the invention preferably at not more than 20% by weight, in particular from 5% by weight to 15% by weight.

In order to adjust the pH to a desired level which does not come about of itself through the mixing of the other components, the compositions of the invention may comprise system-compatible and environmentally compatible acids, especially citric acid, acetic acid, tartaric acid, malic acid, lactic acid, glycolic acid, succinic acid, glutaric acid and/or adipic acid, or else mineral acids, especially sulfuric acid, or bases, especially ammonium hydroxides or alkali metal hydroxides. pH regulators of this kind are present in the compositions of the invention in amounts of preferably not more than 20% by weight, in particular from 1.2% by weight to 17% by weight.

The color transfer inhibitors suitable for use in laundry detergents of the invention include, in particular, polyvinylpyrrolidones, polyvinylimidazoles, polymeric N-oxides such as poly(vinylpyridine N-oxide), and copolymers of vinylpyrrolidone with vinylimidazole.

Graying inhibitors have the function of keeping the soil detached from the textile fiber in suspension in the liquor. Suitable for this purpose are water soluble colloids, usually organic in nature, examples being starch, glue, gelatin, salts of ether carboxylic acids or ether sulfonic acids of starch or of cellulose, or salts of acidic sulfuric esters of cellulose or of starch. Water soluble polyamides containing acidic groups are also suitable for this purpose. Furthermore, starch derivatives other than those mentioned above may be used, aldehyde starches, for example. Preference is given to cellulose ethers, such as carboxymethylcellulose (Na salt), methylcellulose, hydroxyalkylcellulose and mixed ethers, such as methylhydroxyethylcellulose, methylhydroxypropylcellulose, methylcarboxymethylcellulose, and mixtures thereof, in amounts, for example, of from 0.1 to 5% by weight, based on the compositions.

As optical brighteners, laundry detergents of the invention may comprise derivatives of diaminostilbene-disulfonic acid and/or its alkali metal salts. Suitable, for example, are salts of 4,4'-bis(2-anilino-
5 4-morpholino-1,3,5-triazinyl-6-amino)stilbene-2,2'-disulfonic acid or compounds of similar structure which instead of the morpholino group carry a diethanolamino group, a methylamino group, an anilino group, or a 2-methoxyethylamino group. It is also possible for
10 brighteners of the substituted diphenylstyryl type to be present, for example, the alkali metal salts of 4,4'-bis(2-sulfostryl)biphenyl, 4,4'-bis(4-chloro-3-sulfostryl)biphenyl, or 4-(4-chlorostyryl)-4'-(2-sulfostryl)biphenyl. Mixtures of the aforementioned
15 optical brighteners may also be used.

Especially for use in machine processes, it may be of advantage to add customary foam inhibitors to the compositions. Examples of suitable foam inhibitors are
20 soaps of natural or synthetic origin having a high C₁₈-C₂₄ fatty acid fraction. Examples of suitable nonsurfactant-type foam inhibitors are organopolysiloxanes and their mixtures with microfine, optionally silanized silica and also paraffins, waxes,
25 microcrystalline waxes, and mixtures thereof with silanized silica or bis-fatty acid alkylene diamides. With advantages, use is also made of mixtures of different foam inhibitors, for example, mixtures comprising silicones, paraffins, or waxes. The foam
30 inhibitors, especially those containing silicone and/or paraffin, are preferably bound on a granular, water soluble or dispersible support substance. Particular preference is given to mixtures of paraffins and bisstearylethylenediamide.

35 The production of solid compositions of the invention presents no difficulties and may be done conventionally, for example, by spray drying or granulation, with the enzymes and any other heat

sensitive ingredients such as bleaches, for example, being added separately later on if desired. To produce compositions of the invention of heightened bulk density, in particular in the range from 650 g/l to 950 g/l, preference is given to a process known from European Patent EP 0 486 592 which includes an extrusion step. Another preferred preparation, with the aid of a granulation process, is described in European Patent EP 0 642 576.

To produce compositions of the invention in tablet form, which may have one or more phases, may have one or more colors, and consist in particular of one layer or of two or more layers, in particular of two layers, it is preferred to follow a procedure in which all of the constituents - per one layer if appropriate - are mixed with one another in a mixer and the mixture is compressed by means of conventional tableting presses, for example, eccentric presses or rotary presses, at pressing forces in the range from about 50 to 100 kN, preferably from 60 to 70 kN. Especially in the case of multilayer tablets it may be of advantage if at least one layer is compressed beforehand. This is preferably accomplished at pressing forces of between 5 and 20 kN, in particular from 10 to 15 kN. In this way, tablets which are fracture resistant and yet dissolve sufficiently quickly under application conditions are obtained without problems, having fracture strengths and flexural strengths of normally from 100 to 200 N, but preferably above 150 N. A tablet produced in this way preferably has a weight of from 10 g to 50 g, in particular from 15 g to 40 g. The three-dimensional form of the tablets is arbitrary and may be circular, oval or angular, with intermediate forms also being possible. Corners and edges are advantageously rounded. Circular tablets preferably have a diameter of from 30 mm to 40 mm. In particular, the size of tablets of angular or cuboid design which are introduced predominantly by way of the dosing apparatus of, for

example, the dishwashing machine is dependent on the geometry and on the volume of this dosing apparatus. Embodiments which are preferred by way of example have a basal area of (from 20 to 30 mm) × (from 34 to 40 mm), in particular of 26×36 mm or of 24×38 mm.

Liquid or paste detergents of the invention in the form of solutions comprising customary solvents are generally prepared by simply mixing the ingredients, which may be introduced in bulk or in solution into an automatic mixer.

Examples

Example 1

To determine the washing power, cotton fabrics soiled with standardized test stains were washed at 40°C (detergent dosing 76 g; water hardness 16°d [German hardness]; load 3.5 kg, short program) in a domestic washing machine (Miele® W 701). Washing was carried out using a detergent **V1** containing 0.25% by weight of noninventive amylase granules (Termamyl® 60T) and 0.25% by weight of protease granules (activity 200,000 PE/g) containing the Bacillus lentus protease and 18% by weight of sodium percarbonate and 5% by weight of TAED, a detergent **V2** of otherwise the same composition but containing, instead of Termamyl®, the protein-equivalent amount of the genetically modified amylase Duramyl®, a detergent **V3** of otherwise the same composition as **V1** but containing, instead of Termamyl®, the protein-equivalent amount of the genetically modified amylase Purafect® OxAm, a detergent **V4** of otherwise the same composition as **V1** but containing, instead of the Termamyl®, the protein-equivalent amount of the fungal amylase Fungamyl®, and an inventive detergent **M1** of otherwise the same composition as **V1** but containing, instead of Termamyl®, the protein-

equivalent amount of the amylase from *Bacillus amyloliquefaciens*.

The composition of the invention had a wash performance which was markedly superior to that of the compositions containing a different amylase. The situation is essentially the same when the percarbonate in the composition of the invention was replaced by perborate.

10 Example 2

Detergents (**V5** and **V6**, respectively) for the machine washing of kitchen- and tableware, comprising 55% by weight sodium tripolyphosphate (calculated as anhydrous), 4% by weight amorphous sodium disilicate (calculated as anhydrous), 22% by weight sodium carbonate, 9% by weight sodium percarbonate, 2% by weight TAED, 2% by weight nonionic surfactant, and also 1% or 2% by weight amylase granules (Termamyl® 60T) and 1.4% by weight protease granules (activity 200,000 PU/g) comprising the *Bacillus lentus* protease (remainder to 100% by weight water, perfume and dye), compositions (**V7** and **V8**, respectively), whose composition was otherwise like that of **V5** and **V6**, respectively, but which instead of Termamyl® contained protein-equal amounts of Duramyl®, and also compositions in accordance with the invention (**M2** and **M3**, respectively), whose composition was otherwise like that of **V5** and **V6**, respectively, but which instead of Termamyl® contained protein-equal amounts of amylase from *Bacillus amyloliquefaciens*, were tested as indicated below:

In a Miele® G 575 dishwasher (additions of 20 g of each test composition in the universal program, water hardness 14-16°dH [German hardness], operating temperature 55°C), in each case 6 plates soiled with standardized starch stains or bleachable stains were washed and the remaining stain residue was determined

gravimetrically and related to the initial value prior to washing (i.e., 100%). The compositions of the invention were significantly superior to the noninventive compositions in cleaning performance.

- 5 Essentially the same is true if the percarbonate in the compositions of the invention was replaced by perborate.

Annex to our letter of 05.29.2000
PCT/EP99/03563 (H 3491 PCT)

Replacement page

- 27 -

What is claimed is:

1. An amylase containing detergent, characterized in that it comprises α -amylase from *Bacillus amyloliquefaciens* and alkali metal percarbonate in addition to customary ingredients compatible with such constituents, and does not comprise any antibody directed against the amylase.
2. The composition as claimed in claim 1, characterized in that it contains amylase in amounts of from 0.001 mg to 0.5 mg, in particular from 0.02 mg to 0.3 mg, per gram of the overall composition.
3. The composition as claimed in claim 1 or 2, characterized in that it contains up to 50% by weight, in particular from 5% by weight to 30% by weight, of alkali metal percarbonate.
4. The composition as claimed in any of claims 1 to 3, characterized in that it contains from 0.5% by weight to 10% by weight, in particular from 1% by weight to 8% by weight, of compound which under perhydrolysis conditions gives off peroxycarboxylic acid.
5. The composition as claimed in any of claims 1 to 4, characterized in that it contains bleach boosting transition metal salts or transition metal complexes in an amount of up to 1% by weight, in particular from 0.001% by weight to 0.25% by weight.

6. The composition as claimed in any of claims 1 to 5, characterized in that the alkali metal percarbonate comprises sodium percarbonate.

5 7. The composition as claimed in any of claims 1 to 6, characterized in that the alkali metal percarbonate is used in the form of granules prepared and/or coated with the aid of alkaline
10 earth metal sulfate, alkali metal sulfate, alkali metal silicate, alkaline earth metal halide, alkali metal halide, alkali metal carbonate, alkali metal hydrogencarbonate, alkali metal phosphate, alkali metal borate, alkali metal perborate, boric acid, partially hydrated
15 aluminosilicate, carboxylic acids, dicarboxylic acids, polymers of unsaturated carboxylic and/or dicarboxylic acids, or mixtures of these.

20 8. The composition as claimed in any of claims 1 to 7, characterized in that the alkali metal percarbonate has a morphology index of below 0.06.

25 9. The composition as claimed in any of claims 1 to 8, characterized in that it contains from 0.0001 mg to 0.1 mg, in particular from 0.001 mg to 0.01 mg, of α -amylase per percent by weight of alkali metal percarbonate.

30 10. The use of a combination of α -amylase from *Bacillus amyloliquefaciens* and alkali metal percarbonate for enhancing the cleaning performance of detergents which do not comprise any antibody directed against the amylase, when
35 used in washing and cleaning solutions, especially aqueous washing and cleaning solutions.

Replacement page

- 29 -

11. The use as claimed in claim 10, characterized in that the cleaning performance is enhanced in relation to starchy and/or colored stains.

11. The use as claimed in claim 10, characterized in that the cleaning performance is enhanced in relation to starchy and/or colored stains.

"Express Mail" mailing label number EK219527151US

PTO/SB/01 (6-95)

Approved for use through: 10/31/98 OMB 0651-0032

Type a plus sign (+) inside this box → ☐

Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

0010/PTO
Rev. 6/95

U.S. Department of Commerce
Patent and Trademark Office

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

☐ Declaration Submitted with Initial Filing OR ☒ Declaration Submitted after Initial Filing

Attorney Docket Number

H 3491 PCT/US

First Named Inventor

KOTTWITZ, Beatrix

COMPLETE IF KNOWN

Application Number

Filing Date

Group Art Unit

Examiner Name

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

DETERGENT CONTAINING AMYLASE AND PERCARBONATE

(Title of the invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) 05/25/1999 as United States Application Number or PCT International

Application Number PCT/EP99/03563 and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
198 24 688.9	Germany	06/03/1998	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	Additional provisional application numbers are listed on a supplemental priority sheet attached hereto.
		<input type="checkbox"/>

Burden Hour Statement: This form is estimated to take .4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

DECLARATION

Page 2

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code §112.1 acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)
	PCT/EP99/03563	05/25/1999	

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Firm Name Customer Number or label
OR
☒ List Attorney(s) and/or agent(s) name and registration number below:

Name	Registration Number	Name	Registration Number
Wayne C. Jaeschke Glenn E. J. Murphy Stephen D. Harper	21,062 33,539 33,243	See supplemental sheet attached	

☒ Additional attorney(s) and/or agent(s) named on a supplemental sheet attached hereto.

Please direct all correspondence to: ☐ Customer Number or label OR ☒ Fill in correspondence address below

Name	James M. Olsen		
Address	Connolly, Bove, Lodge & Hutz		
Address	P.O. Box 2207 (1220 Market Street)		
City	Wilmington	State	DE
Country	USA	Telephone	302-658-9141
		Fax	302-656-9072

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned

Given Name	Beatrix	Middle Initial		Family Name	KOTTWITZ	Suffix e.g. Jr.	
------------	---------	----------------	--	-------------	----------	-----------------	--

Inventor's Signature Beatrix Kottwitz Date Dec. 12, 2000

Residence: City	Duesseldorf	State		Country	Germany	Citizenship	Germany
-----------------	-------------	-------	--	---------	---------	-------------	---------

Post Office Address Urdenbacher Allee 51

Post Office Address

City	40593 Duesseldorf	State		Zip		Country	Germany	Applicant Authority	
------	-------------------	-------	--	-----	--	---------	---------	---------------------	--

☒ Additional inventors are being named on supplemental sheet(s) attached hereto

DECLARATION

ADDITIONAL INVENTOR(S)
Supplemental Sheet

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

2-00
Given Name **Horst-Dieter** Middle Initial Family Name **SPECKMANN** Suffix e.g. Jr.

Inventor's Signature *Horst-Dieter Speckmann* Date **Dec. 12, 2000**

Residence: City **Langenfeld** State Country **Germany** Citizenship **Germany**

Post Office Address **Eichenfeldstrasse 24 a**

Post Office Address

City **40764 Langenfeld** State Zip Country **Germany** Applicant Authority

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

3-00
Given Name **Karl-Heinz** Middle Initial Family Name **MAURER** Suffix e.g. Jr.

Inventor's Signature *Karl-Heinz Maurer* Date **Dec. 12, 2000**

Residence: City **Erkrath** State Country **Germany** Citizenship **Germany**

Post Office Address **Dechanstrasse 5**

Post Office Address

City **40699 Erkrath** State Zip Country **Germany** Applicant Authority

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

4-00
Given Name **Christian** Middle Initial Family Name **NITSCH** Suffix e.g. Jr.

Inventor's Signature *Christian Nitsch* Date **Dec. 12, 2000**

Residence: City **Duesseldorf** State Country **Germany** Citizenship **Germany**

Post Office Address **Otto-Hahn-Strasse 185**

Post Office Address

City **40591 Duesseldorf** State Zip Country **Germany** Applicant Authority

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

Given Name Middle Initial Family Name Suffix e.g. Jr.

Inventor's Signature Date

Residence: City State Country Citizenship

Post Office Address

Post Office Address

City State Zip Country Applicant Authority

☐ Additional inventors are being named on supplemental sheet(s) attached hereto

DEX

DEX

DEX

Attorney Docket Number: H 3491
First Named Inventor: Beatrix KOTTWITZ
Title: DETERGENT CONTAINING AMYLASE AND
PERCARBONATE

Supplemental Sheet of Additional Attorney(s) and/or Agent(s) :

Connolly, Bove, Lodge & Hutz, of Wilmington, DE 19899

Rudolf E. Hutz	Reg. No. <u>22,397</u>
John D. Fairchild	Reg. No. <u>19,756</u>
Harold Pezzner	Reg. No. <u>22,112</u>
Richard M. Beck	Reg. No. <u>22,580</u>
Paul E. Crawford	Reg. No. <u>24,397</u>
Patricia Smink Rogowski	Reg. No. <u>33,791</u>
Robert G. McMorrow, Jr.	Reg. No. <u>30,962</u>
Ashley I. Pezzner	Reg. No. <u>35,646</u>
William E. McShane	Reg. No. <u>32,707</u>
Mary W. Bourke	Reg. No. <u>30,982</u>
Gerard M. O'Rourke	Reg. No. <u>39,794</u>
James M. Olsen	Reg. No. <u>40,408</u>
Francis DiGiovanni	Reg. No. <u>37,310</u>
Eric J. Evain	Reg. No. <u>42,517</u>
Daniel C. Mulveny	Reg. No. <u>45,897</u>
Elliot C. Mendelson (Agent)	Reg. No. <u>42,878</u>
Christine M. Hansen	Reg. No. <u>40,634</u>